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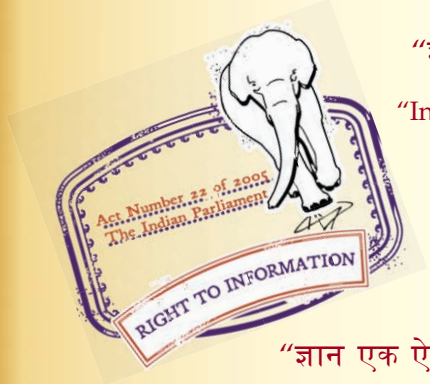
IS 336 (1973): ether [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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"Knowledge is such a treasure which cannot be stolen"

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IS : 336 - 1973
(Reaffirmed 1996)

Indian Standard
SPECIFICATION FOR ETHER
(Second Revision)

Second Reprint OCTOBER 2000

UDC 661.726

BUREAU OF INDIAN STANDARI
MANAK BHAVAN. 9 BAHADUR SHAH ZAFAR MA
NEW DELHI 110002

Gr 6 *January* 1974

AMENDMENT NO. 1 MARCH 2003

TO

IS 336 : 1973 SPECIFICATION FOR ETHER

(Second Revision)

(*Page* 4, *clause* **3.4**, *line* 1) — Substitute '12' *for* '8.5'.

[*Page* 5, *Table* 1, *Sl No.* (i), col 4] — Substitute '0.713 to 0.716' *for* '0.713 0 to 0.714 5'.

(*Page* 7, *clause* **A-2.3**) — Insert the following new clauses after **A-2.3**:

'**A-2.3.1** *General*

A-2.3.1.1 The specified temperature of Relative Density is t_{sp} and the ambient temperature t_{amb} at which Relative Density is conveniently measured, the conversion of the same to that at specified temperature can readily be done by the use of the relationship.

$$\text{Relative Density } (t_{sp}/t_{sp}) = \text{Relative Density } (t_{amb}/t_{amb}) \times [1 \pm |\Delta T| (x_p - x_w)]$$

where

$|\Delta T|$ = difference between t_{sp} and t_{amb} irrespective of sign,

x_p = temperature co-efficient of density of the product, $(dP/dT)_{product}$, and

x_w = $(dP/dT)_{water}$.

A-2.3.1.2 *Use*

Plus sign in Eqn. 1, if $t_{amb} > t_{specified}$ minus sign in Eqn. 1, if $t_{amb} < t_{specified}$. Proof and Examples are given below.

A-2.3.2 *Proof*

$$\text{Relative Density } (t_{sp}/t_{sp}) = \text{Relative Density } (t_{amb}/t_{amb}) \times \frac{1 + \Delta T (dP/dT)_p}{1 + \Delta T (dP/dT)_w} \quad \ldots (1)$$

where the subscripts are:

sp — specified (temp.)

amb — ambient (temp.)

p — product

w — water

$$= \frac{d^{27°C}_{ether}\,[\,1 + \Delta T\,(\,dP/dT\,)_{ether}\,]}{d^{27°C}_{w}\,[\,1 + \Delta T\,(\,dP/dT\,)_{w}\,]}$$

$$= \text{Relative Density (27°/27°C) (as measured)} \times \frac{[\,1 + 7 \times 11.47 \times 10^{-4}\,]}{[\,1 + 7 \times 2.5 \times 10^{-4}\,]}$$

$$= \text{Relative Density (27°/27°C)} \times 1.006\,27$$

Using Eqn. (3), simply,

$$\text{Conversion Factor} = 1 + 7 \times (11.47 - 2.5) \times 10^{-4}$$
$$= 1.006\,28$$

A-2.3.3.2 *When calculated at ambient temperature 15°C*

Ambient temp. = 15°C (let)

$$\text{Relative Density (20°/20°C)} = \text{Relative Density (15°/15°C)} \times \frac{[\,1 - 5 \times 11.47 \times 10^{-4}\,]}{[\,1 - 5 \times 2.5 \times 10^{-4}\,]} =$$

$$\text{Relative Density (15°/15°C)} \times 0.995\,52$$

Using Eqn. (3), simply,

$$\text{Conversion Factor} = 1 - 5 \times (11.47 - 2.5) \times 10^{-4}$$
$$= 0.995\,52$$

(PCD 10)

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Indian Standard

SPECIFICATION FOR ETHER

(Second Revision)

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Indian Standard
SPECIFICATION FOR ETHER
(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 5 June 1973, after the draft finalized by Alcohols and Allied Products Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first published in 1954 and subsequently revised in 1964. The first revision which covers two grades, namely, solvent and anaesthetic grades is further revised in order to reflect the improved quality of the material available in the market. In this revision the anaesthetic grade has been aligned with the corresponding grade prescribed in the Pharmacopoeia of India, Second Edition, 1966. The methods of tests for the determination of peroxides, aldehydes and acetone content have also been modified.

0.3 Ether is extensively used as a solvent in many operations both in industry and the laboratory, including extraction of oils and fats. It is also used extensively as an anaesthetic.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

---

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for ether.

2. GRADES

2.1 The material shall be of two grades, namely, solvent and anaesthetic.

*Rules for rounding off numerical values (*revised*).

3. REQUIREMENTS

3.1 Description — The material shall be a clear, colourless, transparent, highly inflammable, very volatile and mobile liquid.

3.2 Odour and Taste — The material shall possess the characteristic odour and a sweet but burning taste.

3.2.1 *Ether, Anaesthetic* — Pour 10 ml of the material in successive portions on to a clean filter paper and allow to evaporate spontaneously. No foreign odour shall be detectable at any stage of the evaporation.

3.3 Composition

3.3.1 The material shall consist essentially of diethyl ether ($C_2H_5OC_2H_5$).

3.3.2 Ether, solvent or anaesthetic grade, may contain in a proportion not greater than 0·002 percent (*m/v*), a suitable stabilizer, which shall be declared on the label of each container.

3.4 Solubility — The material shall be soluble in about 8·5 volumes of water at 27 ± 2°C and shall be miscible in all proportions with rectified spirit (*see* IS : 323-1959*), with chloroform and with fatty and essential oils.

3.5 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods of test prescribed in Appendix A. References to various clauses of Appendix A are given in col 5 of Table 1.

4. PACKING AND MARKING

4.1 Packing

4.1.1 The material shall be packed in well-closed containers of galvanized iron or glass bottles of amber colour. The packing shall be subject to agreement between the purchaser and the supplier and to the provisions of law in force in the country for the time being.

4.1.2 All containers in which the material is stored shall be clean, dry and leak-proof. The containers shall be protected from light and stored in a cool place. If the containers are closed with corks, they shall be protected with metal foils and suitably secured.

4.2 Marking

4.2.1 Each container shall be marked with the following:

a) Name and grade of the material;

*Specification for rectified spirit (*revised*).

b) Name of the manufacturer, initials or his trade-mark, if any;

c) Net, gross and tare mass;

d) Month and year of manufacture; and

e) Name and proportion of stabilizer added.

TABLE 1 REQUIREMENTS FOR ETHER

(Clause 3.5)

Sl No.	Characteristic	Requirement		Method of Test (Ref to Cl No. in Appendix A)
		Solvent Grade	Anaesthetic Grade	
(1)	(2)	(3)	(4)	(5)
i)	Relative density at 20°C/20°C	0·713 to 0·718	0·713 0 to 0·714 5	A-2
ii)	Peroxides	Shall pass the test		A-3
iii)	Distillation range	Entire volume shall distil within the range of 34 to 36°C, the temperature being corrected* for a pressure of 760 mm Hg	Entire volume shall distil within the range of 34 to 35°C, the temperature being corrected* for a pressure of 760 mm Hg	A-4
iv)	Residue on evaporation, g/100 ml, *Max*	0·002 (excluding added stabilizers)	0·002 (excluding added stabilizers)	A-5
v)	Methyl alcohol	Shall pass the test		A-6
vi)	Aldehydes and acetone	do		A-7
vii)	Sulphurous acid and other free acids	do		A-8

*In case the barometric pressure, p, deviates from 760 mm Hg, use the correction 0·037 (p-760)°C for the distillation temperature.

4.2.2 Each container shall have the caution label 'FLAMMABLE' together with the corresponding symbol for labelling of dangerous goods as given in Fig. 3 of IS : 1260-1958*.

Note 1 — Necessary safeguards against the risk arising from storage and handling of large volumes of flammable liquids shall be provided and all due precautions shall be taken at all times to prevent accidents by fire or explosion.

Note 2 — Except when they are opened for the purpose of cleaning and rendering them free from ether vapours, all empty tanks or other containers shall be kept securely closed unless they have been cleaned and freed from ether vapours.

*Code of symbols for labelling of dangerous goods.

4.2.3 The product may also be marked with Standard Mark.

4.2.4 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manfucaturers or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 The method of drawing representative samples of the material and criteria for conformity shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 3.5 and *Table* 1)

METHODS OF TEST FOR ETHER

A-0. QUALITY OF REAGENTS

A-0.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070 - 1960*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-1. GENERAL

A-1.1 All tests shall be carried out, as far as possible, within 5 days of sampling. After opening the glass bottle containing the sample, tests shall be carried out as quickly as possible so as not to expose the material unduly to the action of air.

A-1.2 Fire and Other Hazards — A mixture of two percent of ether vapour and air is flammable, or is explosive if confined or localized. *Special care, therefore, shall be taken in the storage of this material and carrying out the test for range of distillation and residue on evaporation.*

*Specification for water, distilled quality (*revised*).

A-2. DETERMINATION OF RELATIVE DENSITY

A-2.1 Apparatus

A-2.1.1 *Relative Density Bottle* — Stoppered, Regnault type, of not less than 25 ml capacity, and provided with a ground-glass capillary-hole stopper having a graduation mark on the stopper.

A-2.2 Procedure — Clean, dry and weigh the relative density bottle and the stopper. Fill the bottle with water and immerse it up to the neck in a constant temperature bath at about 18°C for 20 minutes. Allow the temperature of the bath to rise to 20·0 ± 0·2°C and maintain this temperature for 20 minutes. Suck off the water with a bit of filter paper till the level reaches the graduation mark and weigh again. Empty the bottle, clean and dry. Repeat the operation with the material at 20°C.

A-2.3 Calculation

$$\text{Relative density at } 20°C/20°C = \frac{A - B}{C - B}$$

where

A = mass in g of the relative density bottle with the material at 20°C,

B = mass in g of the relative density bottle, and

C = mass in g of the relative density bottle with water at 20°C.

A-3. TEST FOR PEROXIDES

A-3.1 Reagents

A-3.1.1 *Potassium Iodide* — iodate free.

A-3.1.2 *Potassium Iodide Solution* (*For Solvent Grade*) — Dissolve 10 g of potassium iodide in sufficient water to produce 95 ml of solution.

A-3.1.3 *Potassium Iodide and Starch Solution* (*For Anaesthetic Grade*) — Dissolve 10 g of potassium iodide in sufficient water to produce 95 ml of solution. Add 5 ml of starch solution (**A-3.1.9**). This solution shall be freshly prepared.

A-3.1.4 *Iodine* — resublimed.

A-3.1.5 *Arsenic Trioxide* — pure, previously dried to constant mass at 105°C.

A-3.1.6 *Sodium Hydroxide Solution* — approximately 1 N.

A-3.1.7 *Hydrochloric Acid* — approximately 1 N.

A-3.1.8 *Sodium Bicarbonate Solution* — saturated.

A-3.1.9 *Starch Solution* — Triturate 0·5 g of starch with 5 ml of water and add this, with constant stirring, to sufficient boiling water to produce 100 ml.

Boil for 3 minutes, allow the solution to cool and when the sediment has settled, decant off the clear liquid. If necessary, filter. This solution shall be freshly prepared.

A-3.1.10 *Standard Iodine Solution* — 0·001 N. Weigh 12·8 to 12·9 g of resublimed iodine, mix it with 18 g of potassium iodide, add 30 ml of water and allow the whole to stand until the solution of the iodine is complete. Dilute the solution with water to 1 000 ml and standardize it against pure dry arsenic trioxide. For this, transfer about 0·42 to 0·45 g of arsenic trioxide, accurately weighed, into a 500-ml conical flask, add 20 ml of sodium hydroxide solution and warm the flask to aid the solution. Cool to room temperature, add 15 ml of hydrochloric acid to neutralize the excess of alkali. Dilute to 200 ml and add 20 ml of sodium bicarbonate solution. Titrate the solution with iodine solution, adding 2 to 3 ml of starch solution as indicator towards the end of the titration, until a pale purple pink colour is obtained throughout the solution. Knowing the strength of this iodine solution, freshly prepare exactly 0·1 N iodine solution by accurate dilution of standardized iodine solution using the following formula:

$$V = 10r$$

where

V = the volume in ml to which 1 ml of the iodine solution has to be diluted to make it exactly 0·1 N, and

r = the normality of the standard iodine solution.

Pipette out exactly 10 ml of the 0·1 N solution into a measuring flask, dilute with water and make up the volume to 1 000 ml. The resulting solution is 0·001 N.

A-3.2 Procedure for Ether, Solvent — Place 8 ml of freshly prepared potassium iodide solution (**A-3.1.2**) in a glass-stoppered tube of about 12 ml capacity (*see* Note) and about 15 mm diameter. Fill to the brim with a portion of ether, solvent, being tested. Place the stopper in position so that no air bubble is enclosed, shake vigorously, and set aside in the dark for 30 minutes.

A-3.2.1 The material shall be taken to have satisfied the requirements if the yellow colour produced in either layer, if any, is not deeper than that of 0·5 ml of freshly prepared 0·001 N iodine solution diluted with 8 ml of potassium iodide solution.

NOTE — In case 12 ml capacity tube is not available, then 10 ml capacity tube may be used and correspondingly the proportions of reagents should be adjusted.

A-3.3 Procedure for Ether, Anaesthetic — Place 8 ml of potassium iodide and starch solution (**A-3.1.3**) in a glass-stoppered tube of about 12 ml capacity (*see* Note) and about 15 mm diameter. Fill to the brim with

a portion of ether, anaesthetic, being tested. Place the stopper in position so that no air bubble is enclosed, shake vigorously, and set aside in the dark for 30 minutes.

A-3.3.1 The material shall be taken to have passed the test if no brown or reddish colour is produced in either layer.

NOTE — In case 12 ml capacity tube is not available, then 10 ml capacity tube may be used and correspondingly the proportions of reagents should be adjusted.

A-4. DETERMINATION OF DISTILLATION RANGE

A-4.0 Caution — Ether of high peroxide content is liable to explode on heating to boiling temperature. The test for peroxide (*see* **A-3**) shall, therefore, be performed before proceeding to distillation of the material. It shall always be ensured that peroxides are absent (*see* **A-3**) before commencing this determination.

NOTE — The test shall be carried out at a temperature less than 27°C, ambient.

A-4.1 Apparatus

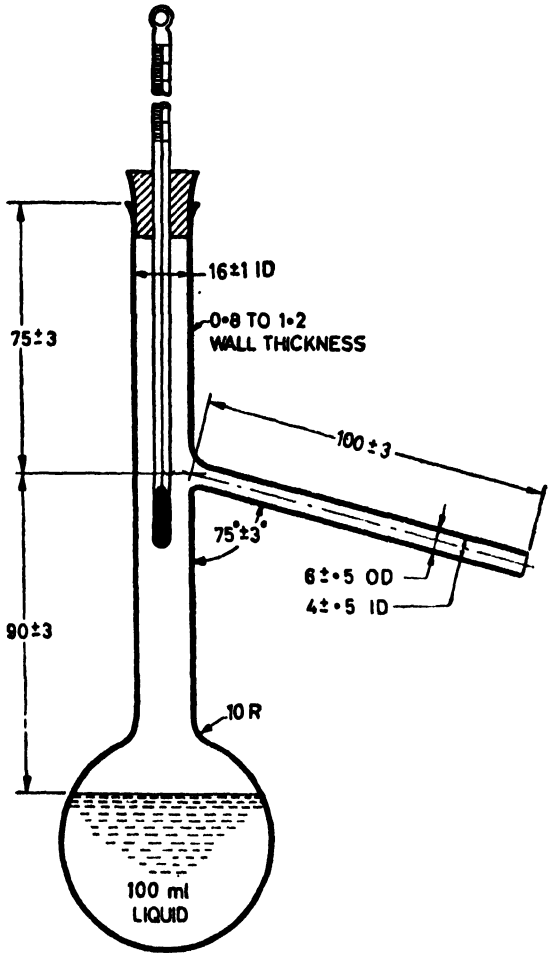
A-4.1.1 *Distillation Flask* — of the shape and dimensions given in Fig. 1.

A-4.1.2 *Thermometer** — Conforming to the following requirements is recommended:

Range	2 to 80°C
Graduation	0·2°C
Immersion	Total
Overall length	390 mm
Stem diameter	5·5 to 8·0 mm
Bulb shape	Cylindrical
Bulb length	9 to 14 mm
Bulb diameter	4·5 to 5·5 mm
Length of graduated portion	245 to 280 mm
Distance from bottom of bulb to bottom of main scale	75 to 90 mm
Longer lines at each	1°C
Figured at each	2°C
Expansion chamber	To allow heating to 130°C
Top finish	Ring
Scale error not to exceed	±0·2°C

A-4.1.2.1 Any other thermometer of similar range and accuracy may also be used.

*Thermometers with the Institute of Petroleum designation IP 60C conform to these requirements.



All dimensions in millimetres.

FIG. 1 DISTILLATION FLASK (CAPACITY 130 ±5 ml)

A-4.1.2.2 The thermometer shall bear a certificate from the National Physical Laboratory (CSIR), New Delhi, or any other institution authorized by the Government of India to issue such a certificate.

A-4.1.3 *Draught Screen* — The construction and dimensions of the draught screen shall be as shown in Fig. 2. A shelf of heat-resisting hard asbestos

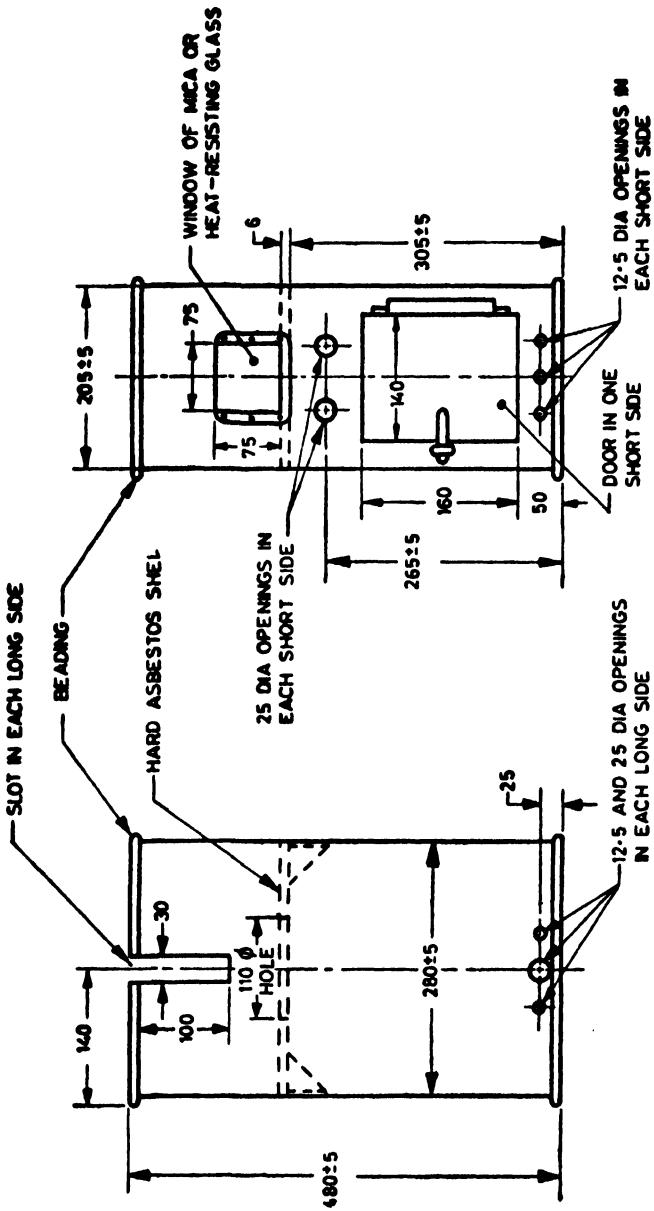


FIG. 2 DRAUGHT SCREEN

compound, 6 mm in thickness with a central circular hole of 110 mm in diameter, the edge of which is levelled to fit the contours of the distillation flask, shall be supported horizontally in the screen and shall fit closely to the sides of the screen to ensure that hot gases from the source of heat do not come in contact with the sides or neck of the flask. The supports for this asbestos shelf may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners. In each of the longer sides of the screen there shall be one large and two small holes at the base, and a vertical slot at the top. A removable shutter conforming to the dimensions shown in Fig. 3 shall be provided for closing whichever vertical slot is not in use. In each of the shorter sides of the screen there shall be two circular holes below the asbestos shelf, three circular holes at the base, and a central window of mica or heat-resisting glass, the bottom of which shall be levelled with the top of the asbestos shelf. In one of the shorter sides there shall also be a door, overlapping an opening in the screen by approximately 5 mm, all round.

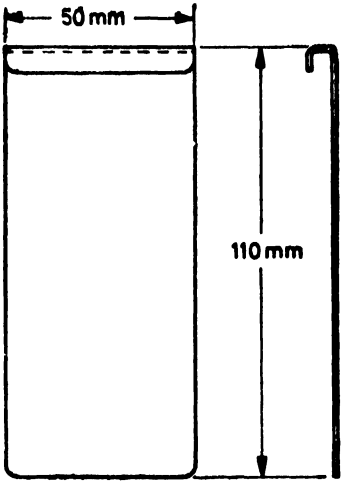


FIG. 3 REMOVABLE SHUTTER FOR DRAUGHT SCREEN

A-4.1.4 *Condenser and Cooling Bath* — A suitable condenser may be used but in case of dispute a condenser as shown in Fig. 4 shall be used. The condenser tube shall be a seamless brass tube, having a wall thickness of 1·2 mm. The tube and bath shall conform to the following dimensions:

Outside diameter of tube	14·3 ± 0·5 mm
Overall length of tube	560 ± 5 mm
Length of tube within bath	Approximately 390 mm
Length of tube projecting from bath at upper end	Approximately 50 mm
Length of tube projecting from bath at lower end	Approximately 115 mm
Length of shorter limb of tube	Approximately 76 mm
Angle included between longer and shorter limb	97 ± 3°
Length of bath	380 ± 5 mm
Width of bath	100 ± 5 mm
Height of bath	150 ± 5 mm
Distance from top of bath to centre line of tube at point of entrance	Approximately 30 mm
Distance from bottom of bath to centre line of tube at point of exit	Approximately 20 mm

The cooling bath shall be provided with a tap at the bottom for drainage or inlet, with an overflow tube near the top, and with a suitable stand such that the base of the cooling bath is approximately 300 mm above the bench level.

NOTE — Small variations in the height of the side tube of the distillation flask above bench level are unavoidable. It is, therefore, desirable that the legs of the stand be made of malleable iron, so that they may be bent to bring the condenser tube into alignment with the side tube of the distillation flask. Alternatively, alignment may be achieved by standing either the draught screen or the condenser in a board of suitable thickness.

A-4.1.5 *Water-Bath* — with a steam coil, electrical immersion heater or other suitable heating device (other than naked flame) for maintaining the temperature of water.

A-4.1.6 *Receiver* — a 100-ml graduated cylinder, with 1 ml marks running halfway round the circumference, with 5 ml marks running three-quarters-way round and with 10 ml marks running all round the circumference and numbered.

A-4.2 Assembly of Apparatus — Assemble the apparatus as shown in Fig. 5 giving particular attention to the following points:

a) *Position of Thermometer* — The thermometer shall be held concentrically in the neck of the flask by means of a well fitting cork, and the lower end of the main capillary tube shall be in level with the highest point of the bottom of the bore of the side-tube (*see* Fig. 6). The cork shall project about 10 mm above the top of the neck of the flask.

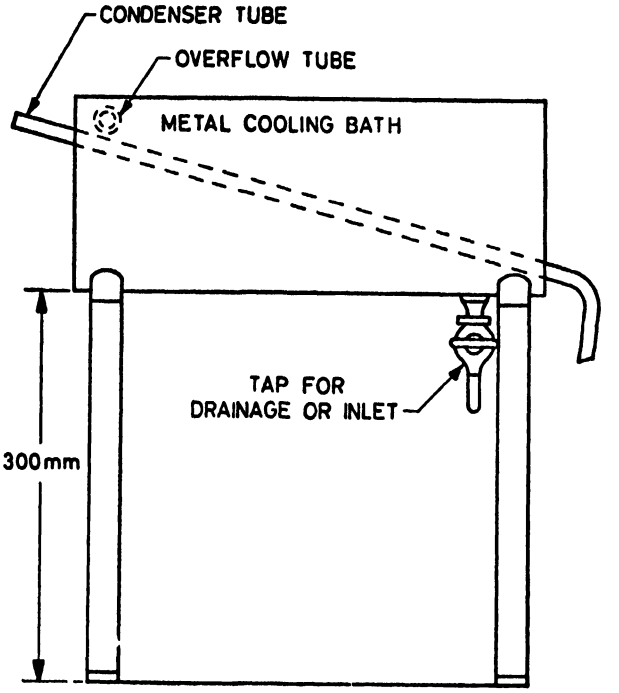


FIG. 4 CONDENSER

b) *Support for Flask* — When an asbestos board is used to support the flask, the one having a central hole of size appropriate to the particular distillation in hand shall be placed on top of the asbestos shelf in the draught screen so that the holes are approximately concentric. The flask shall be placed in position and pressed down so as to close completely the hole in the asbestos board. In some distillations a wire gauze is specified for supporting the flask in place of the asbestos board.

c) *Connection of Flask to Condenser* — The flask shall be so connected to the condenser that the end of the side-tube projects at least 25 mm beyond the cork into the condenser.

A-4.3 Procedure — Fill the condenser cooling bath with water at a temperature of 5 ± 2°C and immerse the receiver up to the 90 ml mark in a bath of water maintained at the same temperature. Place the water-bath on its support so that it is flush with the underside of the shelf of the draught screen and fill it to within 3 mm of the top with water at 60°C. Measure in the receiver 100 ml of ether previously brought to the temperature of the

condenser water, transfer as completely as possible to the distillation flask and add a few pieces of clean, dry porous pot. Place the flask, thermometers and receiver in position, close the neck of the receiver with a plug of cotton wool and ensure, by addition of ice, that the condenser cooling bath remains at a temperature of 5 ± 2°C. Regulate the supply of heat to the water-bath so that the temperature of the water is maintained at 60 ± 2°C throughout the distillation. This should ensure a distillation rate of 3 to 4 ml per-minute. Read the volume of distillate in the receiver when the flask thermometer just reaches either, (a) the corrected specified distillation temperature or, (b) the maximum temperature of the distillation, whichever is lower.

A-4.3.1 If the volume of the distillate is less than 90 ml, repeat the distillation, keeping the receiver, suitably weighted, in ice-cold water.

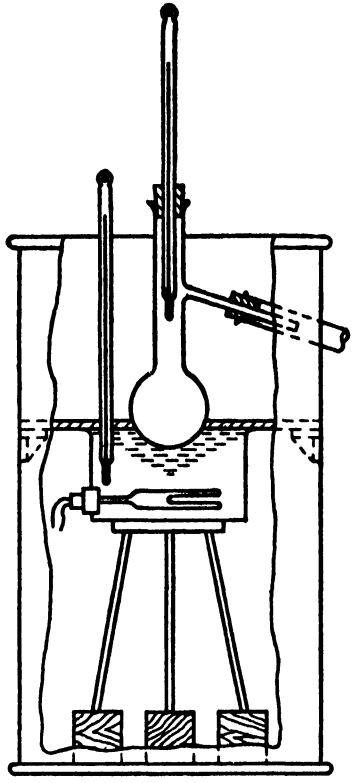


FIG. 5 ASSEMBLY OF APPARATUS

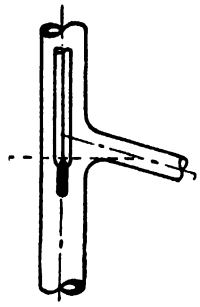


FIG. 6 POSITION OF THERMOMETER IN DISTILLATION FLASK

A-4.4 Correction of the Thermometric and Barometric Readings

A-4.4.1 *Error of Scale* — In all thermometer readings, make the corrections as indicated on the certificate of the instrument.

A-4.4.2 *Correction for Barometric Pressure* — In case the barometric pressure, p, deviates from 760 mm Hg, use the correction as given in the foot-note in Table 1.

A-5. DETERMINATION OF RESIDUE ON EVAPORATION

A-5.0 Caution — Ether of high peroxide content is liable to explode on heating to boiling temperature. The test for peroxide (*see* **A-3**) shall, therefore, be performed before determining the residue on evaporation. If the determination of residue on evaporation has to be carried out on ether of high peroxide content, suitable precautions shall be taken during heating. When evaporating ether, it is advisable to cover the dish on the water-bath with a large funnel and remove the vapours rapidly by vacuum. The vapours may be aspirated through water in a large (10 litre) bottle. It shall, however, always be ensured that the peroxides are absent before commencing this determination.

A-5.1 Procedure — Evaporate 100 ml of ether to dryness in a weighed platinum, silica or borosilicate glass basin on a water-bath. Dry the residue for 30 minutes in an oven at a temperature of 100 $\pm$ 2°C. Cool in a desiccator and weigh. Repeat till constant mass is obtained. Report this mass as number of grammes per 100 ml.

A-6. TEST FOR METHYL ALCOHOL

A-6.1 Reagents

A-6.1.1 *Solution of Potassium Permanganate in Orthophosphoric Acid* — Dissolve 3·0 g of potassium permanganate in a mixture of 15 ml of orthophosphoric acid and 70 ml of water. Add sufficient water to make up to 100 ml.

A-6.1.2 *Dilute Sulphuric Acid* — 1 : 1 (*v/v*).

A-6.1.3 *Solution of Oxalic Acid in Dilute Sulphuric Acid* — Dissolve 5·0 g of oxalic acid in dilute sulphuric acid and add sufficient dilute sulphuric acid to make up to 100 ml.

A-6.1.4 *Sodium Sulphite* — solid (*see* IS : 247-1964*).

A-6.1.5 *Concentrated Hydrochloric Acid* — Relative density 1·16 (*see* IS : 265-1962†).

A-6.1.6 *Decolourized Solution of Magenta* — Dissolve 1·0 g of basic magenta (fuchsin) in 600 ml of water and cool it in an ice-bath. Add 20 g of sodium sulphite dissolved in 100 ml of water, cool in an ice-bath and add, slowly and with constant stirring, 10 ml of concentrated hydrochloric acid. Dilute to 1 000 ml. Filter the solution, if turbid.

A-6.1.6.1 Protect the decolourized solution of magenta from light. If the solution is brown in colour, shake it with sufficient animal charcoal (0·2 to 0·3 g) to render it colourless and then filter immediately. Occasionally, it is necessary to add 2 to 3 ml of hydrochloric acid followed by shaking to remove a little residual pink colour. Before using, allow the solution resulting from any of the foregoing treatments to stand overnight.

A-6.1.7 *Dilute Ethyl Alcohol* — Obtained by diluting 210 ml of rectified spirit (conforming to IS : 323-1959‡) to 1 000 ml with water.

A-6.2 Procedure — Shake vigorously 10 ml of ether with 5 ml of dilute ethyl alcohol and 5 ml of water in a separating funnel. Allow the mixture to separate, and draw off the lower layer which will contain any methyl alcohol present in the material. To 5 ml of this solution add 2·0 ml of a solution of potassium permanganate in orthophosphoric acid. Leave it for 10 minutes, add 2·0 ml of a solution of oxalic acid in sulphuric acid, and then add 5 ml of decolourized solution of magenta. Leave it at a temperature of 15 to 30°C and examine after 30 minutes.

A-6.2.1 The material shall be taken to have passed the test, if no colour is developed.

A-7. TEST FOR ALDEHYDES AND ACETONE

A-7.0 General — This test is apt to be vitiated, if a stabilizer, such as hydroquinone is present. It is recommended that the test may be carried on the distillate obtained by distilling the material carefully in a fractionating column.

*Specification for sodium sulphite, anhydrous (*second revision*).

†Specification for hydrochloric acid (*revised*).

‡Specification for rectified spirit (*revised*).

A-7.1 Reagents

A-7.1.1 *Potassium Iodide* — solid.

A-7.1.2 *Mercuric Chloride Solution* — saturated aqueous solution.

A-7.1.3 *Sodium Hydroxide* — fused solid.

A-7.1.4 *Nessler's Solution* — Dissolve 10 g of potassium iodide in 10 ml of ammonia-free water, and add to it, slowly with stirring, mercuric chloride solution until a slight permanent precipitate forms. Add 30 g of sodium hydroxide and, when it has dissolved, add 1 ml more of mercuric chloride solution and dilute to 200 ml with ammonia-free water. Allow to settle overnight, decant off the clear solution and keep the solution in a bottle closed with a well-fitting rubber stopper.

A-7.2 Procedure for Ether, Anaesthetic — Place in a glass-stoppered tube, of about 12 ml capacity and about 15 mm diameter, 2 ml of Nessler's solution and fill the tube with a portion of the material, insert the stopper, shake vigorously for ten seconds, and leave it for 5 minutes.

A-7.2.1 The material shall be taken to have passed the test, if no colour or turbidity is produced.

A-7.2.2 A very faint opalescence may sometimes be produced, but there shall be no colouration or colouration followed by turbidity.

A-7.2.3 If hydroquinone or some other stabilizer is stated to be present and some colour or turbidity is produced in the above test, repeat the test after distilling the material in a fractionating column.

A-7.3 Procedure for Ether, Solvent — Shake 10 ml of the material occasionally during 2 hours with 1 ml of potassium hydroxide solution (containing 4·75 to 5·25 percent *m/v* of KOH), in a glass-stoppered cylinder of colourless glass protected from light.

A-7.3.1 The material shall be taken to have passed the test, if no colour is produced in either layer.

A-8. SULPHUROUS ACID AND OTHER FREE ACIDS

A-8.1 Reagents

A-8.1.1 *Phenolphthalein Indicator* — Dissolve 0·1 g of phenolphthalein in 100 ml of 60 percent rectified spirit.

A-8.1.2 *Alcohol* — ethyl alcohol 80 percent (*v/v*).

A-8.1.3 *Standard Sodium Hydroxide Solution* — 0·02 N.

A-8.2 Procedure — Place 10 ml of ethyl alcohol in a 50-ml glass-stoppered flask, add 0·5 ml of solution of phenolphthalein indicator and just sufficient

sodium hydroxide solution to produce a pink colour which persists after shaking the mixture for 30 seconds. Add 25 ml of the material, mix gently and add sodium hydroxide solution until the pink colour persists after shaking the mixture for 30 seconds.

A-8.2.1 The material shall be taken to have passed the test, if not more than 0·4 ml of additional standard sodium hydroxide solution is required in case of ether, solvent, and 0·2 ml of additional standard sodium hydroxide solution in case of ether, anaesthetic, to turn the solution pink.

APPENDIX B

(*Clause* 5.1)

SAMPLING OF ETHER

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall be taken in a protected area with good ventilation.

B-1.2 Sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking, stirring, rolling or by any other suitable means.

B-1.5 The samples shall be placed in suitable, clean, dry and air-tight amber-coloured glass containers protected from light by wrapping with a black paper.

B-1.6 The sample containers shall be of such a size that an ullage of about 5 percent is left after pouring in the sample.

NOTE — Containers, capable of holding about 400 and 300 ml of material may generally be adequate in practice.

B-1.7 Each sample container shall be sealed air-tight with a suitable stopper after filling, and marked with the manufacturer's name or trade-mark, the month and year of manufacture of the material, the batch number (if available) and other details of sampling, such as the date of sampling, sampler's name, etc.

B-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

B-2. SAMPLING INSTRUMENT

B-2.1 It is made of thick glass and is 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 7). The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For drawing sample, the apparatus is first closed at the top with the thumb or a stopper and lowered till a desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

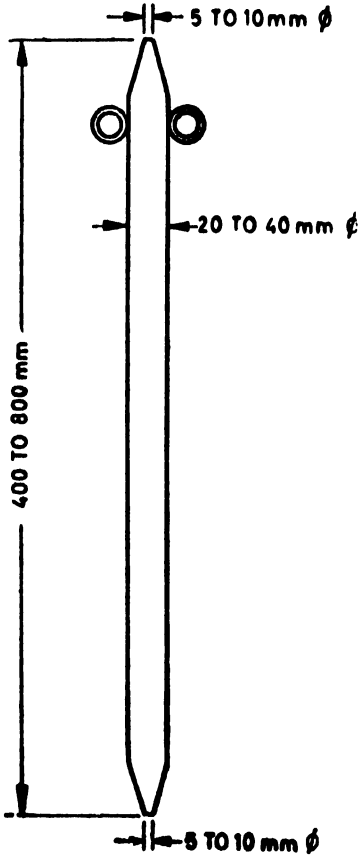


FIG. 7 SAMPLING TUBE

B-2.1.1 For small containers, the size of the sampling tube may be altered suitably.

B-3. SCALE OF SAMPLING

B-3.1 Lot — In a single consignment of one grade of material, all the containers of the same size and drawn from the same batch of manufacture shall constitute a lot. If a consignment of one grade of material is known to consist of different batches of manufacture in different sizes of containers, the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

B-3.2 For ascertaining the conformity of the material in a lot to the requirements of this specification, tests shall be carried out for each lot separately. For this purpose 5 containers shall be selected at random from each lot.

NOTE — In the case of very small lots where the selection of 5 containers may be uneconomical, th number of containers to be selected and the method of judging the conformity of the lot to the requirements of the specification shall be as agreed to between the purchaser and the supplier.

B-3.3 The containers shall be selected at random and to ensure randomness of selection, some random number table as agreed to between the purchaser and the supplier shall be used. In case such a table is not available, the following procedure shall be adopted:

Starting from any container in the lot, count them as 1, 2,....., up to r and so on in one order, where r is the integral part of $N/5$ (N being the number of containers in the lot). Every rth container thus counted shall be withdrawn to constitute a sample till the required number of 5 containers is obtained.

B-4. PREPARATION OF TEST SAMPLES

B-4.1 From each of the containers selected according to **B-3.3**, a portion of the material not less than 1 000 ml shall be drawn with the help of the suitable sampling instrument (*see* **B-2**).

B-4.2 Out of these portions, a small but equal quantity of material shall be taken and mixed thoroughly to form a composite sample, of about 1 000 ml. The composite test sample shall be divided into 3 equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-4.3 The remaining portion of the material drawn from each container shall be divided into 3 equal parts each forming an individual sample. One set of individual samples, representing the 5 containers sampled, shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

B-4.4 All the individual and composite samples shall be transferred to separate containers and shall be sealed and marked with full identification particulars given under **B-1.7.**

B-4.5 The referee test samples consisting of a composite sample and a set of 5 individual samples shall bear the seal of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier, to be used in the case of any dispute between the two.

B-5. NUMBER OF TESTS

B-5.1 Tests for the determination of distillation range shall be conducted on each of the individual samples (*see* **B-4.3**).

B-5.2 Tests for the determination of all other characteristics given in **3** shall be conducted on the composite sample (*see* **B-4.2**).

B-6. CRITERIA FOR CONFORMITY

B-6.1 For Individual Samples — The lot shall be declared as conforming to the requirements of distillation range if each of the individual test results satisfies the relevant requirement given in Table 1, Item (iii).

B-6.2 For Composite Sample — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample (*see* **B-5.2**), the test results for each of the characteristics shall satisfy the relevant requirement given in this specification.

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Printed at New India Printing Press, Khurja, India